Environmental Technology Center

CIBA-GEIGY Corporation
P.O. Box 18300
410 Swing Road
Greensboro, North Carolina 27419
Telephone 919 632 6000
Fax 919 632 2048

CIBA-GEIGY

FAXED TO EPA
3-19-91
F.B.

REC'D 3-25-91
F.B.

March 18, 1991

Ms. Deb Szarro
UPEPA Region I
60 Westview Street
Lexington, MA 02173

Dear Ms. Szarro

As follow-up to our phone conversations on 3/15, here is a summary of events that took place regarding the dioxin Performance Evaluation (PE) Samples included in the Cranston project and additional discussion regarding appropriate laboratory actions to correct the problem.

Summary of Events:

- I notified you verbally of the validated results obtained by Radian for the PE samples mid-morning.
- You left me voicemail saying that one PE was "100% unacceptable".
- We discussed the specifics of the results of the PE samples: CIJ993 detected nothing and was acceptable, ZH0115 detected 1.7 ppb 2,3,7,8-TCDD and 6.8 ppb of other TCDD isomers which gives a total TCDD concentration of 8.5 ppb. The reported results of sample ZH0115 were unacceptable according to the Region I information. The basis of the rejection was reporting false positives.
- Our discussion then turned to possible lab corrective actions. Since the focus of the error was on false positives, the integrity of the data for samples which were reported to contain 2,3,7,8-TCDD are in question. Reanalysis of these samples along with reanalysis of both PE samples was mutually agreed to be an appropriate action by the lab.
- Since the code of the bottle ZH0115 was incorrectly reported to me as ZN0115 by the data validator, I contacted our contractor (Mark Houlday of Woodward Clyde) to have the data validator re-verify the results reported to me to ensure there were no other typos. The validator (Nancy Potak) confirmed that during validation she verified as accurate all of the results for this sample. There was no need to change the reported concentrations from Radian during validation. She sent a copy of the supporting documentation to me. The percent valley of the continuing calibration associated with the PE was <9%, so reporting a non-2,3,7,8-TCDD isomer as 2,3,7,8-TCDD should not have occurred during the analysis.

- Concurrently, Radian investigated the PE results. They recalculated their results, re-injected the extract and confirmed their original results. The following anomalies were detected, but they do not affect the results in question.

    - The entire sample in the jar (14 gm) was extracted. Since the results are reported in ppb and the weight of the original sample is in the calculation, there is no affect in the final, reported concentration.

    - The reported detection limits for sample ZH0115 were not adjusted for the 14 gm sample size. The detection limits were reported as 2.2 ppb; actually Radian calculated the value 2.2 in nanograms (ng) and omitted the weight adjustment. The corrected detection limit is 0.15 ppb as calculated by Radian.

- I left voicemail for you stating that the results were reconfirmed and that I would send a fax with the values written to insure I did not mis-speak the values we intended to report.

- I left voicemail for Frank Battiglia informing him of the PE sample issue and that we are in the process of resolving it.

- We await either confirmation that the results are OK or re-confirmation that the results are unacceptable.

Additional Discussion:

If I assume that the written results will be the same as the verbal results and that there was no error on your part verifying the acceptance windows, then the rejection by Region I of the Radian PE results will stand. Since I coordinated the dioxin PE samples for Region III for several years, and know the value of including them with each "batch" of samples, I had requested them through Frank Battaglia for the Cranston project. As you know, there are various types of dioxin PE samples and each type provides different information regarding the quality of the data.

After giving Radian's data considerable thought, I have come up with two possible scenarios for failure:

Possible reason for failure #1: There was no 2,3,7,8-TCDD present and the lab reported 1.7 ppb.

If this is the reason for failure, all of the reported positives are in jeopardy.

Possible reason for failure #2: The PE sample contained a low level concentration of 2,3,7,8-TCDD and the reported concentration is outside the EPA 99% Confidence Interval.

If this is the reason for failure, then the reported results

verify Radian can detect low levels of 2,3,7,8-TCDD (all of the identification criteria were met). It may also show that the reported positives for 2,3,7,8-TCDD in other samples in the Cranston project may be biased high.

In order to proceed in both a technically appropriate and timely manner, I need to know more information regarding the reason for failure. I fully understand that it is not in either Region I nor CIBA-GEIGY's best interest to fully explain the exact reason for failure, if that information reaches the lab and incomplete corrective action is taken. I have notified Radian that they are not to contact Region I regarding this issue, and that all communication will flow through me to you.

Requested Region I Follow-up:

If reason #2 is the one which applies to ZH0115, please provide the following:

- Please verify that this PE is intended to be used quantitatively for Method 8280. Based on my prior discussions with EMSL-LV (as an EPA employee), some PE samples were intended to be used for quantitative assessment of only Method 8290 performance where the detection limits are in the parts per trillion range.

- Please verify or refute whether the other (3?) PE samples from Region I that have recently failed were related to the same lot as ZH0115.

- Please verify through Larry Butler that the acceptance limits provided by EMSL-LV for sample ZH0115 are based on actual analysis and are not predicted windows which are untested.

- Please verify or refute whether either of the PE samples sent to Radian with Round 2 of Phase 1B are appropriate to indicate whether the lab is now in control for the error encountered in Round 1. If the PE samples currently being analyzed by Radian are appropriate, then no additional PE samples may be needed. If, however, the current PE samples are significantly different, please make an additional pair of PE samples available that are similar to the ones in Round 1. As noted earlier, Radian analyzed the entire sample in the PE sample jar, so there is none left to reanalyze to show that they have corrected their source of error.

CIBA-GEIGY Follow-up:

The appropriate corrective action will be based on your response regarding the reason for rejection and verification of the integrity of the PE lot (i.e., EMSL-LV has certified results for this lot and no other unresolved problems have been reported).

I look forward to our next communications, a timely resolution of this issue, and generation accurate and reliable results for dioxin in Round 2. Please contact me at your earliest opportunity so that resolution of this issue can begin immediately. In the event that I cannot be reached, please contact either Diane Leber (x2159) or Jim Crowley (x2196) at 914 479-5000.

Sincerely,

Diana Baldi

Diana Baldi, Administrator
National Service Contracts

cc: Ken Dupuis, TRP
Jim Crowley, Ardsley
Diane Leber, Ardsley
Mark Houlday, Woodward Clyde
Frank Battaglia, USEPA Region I
Kelly Young, Radian

THE UNIVERSITY OF MICHIGAN
School of Natural Resources

AMERICAN HOESCHT NPDES COMPLIANCE MONITORING DATA

- WILL WITNESS RIVER SEDIMENT VOLS [illegible] - ALLIANCE CAN DO SPLITS?
- SEE CHARLIE PORFERT

TO PETER NORAN ON 3/14/91 AM

FINAL COPY OF VALID WORKSHEETS

SEE ABOV MORE CONTRACT #354

KATHY LIOSSA 504 765 0358

30% SOLID FOR SEDIMENTS

JUDGEMENT CALL

CORE 6" →

VOLATILES DO NOT MIX

STEVE STODOLA 860-7634

BLANK & SPIKE

NONVOL MIX & TAKE SPLITS

[illegible] $312

~~IN ALLIANCE~~ FINAL REPORT MENTION VISUALS ON SAMPLES

Dana Building 430 E. University
Ann Arbor, Michigan 48109-1115